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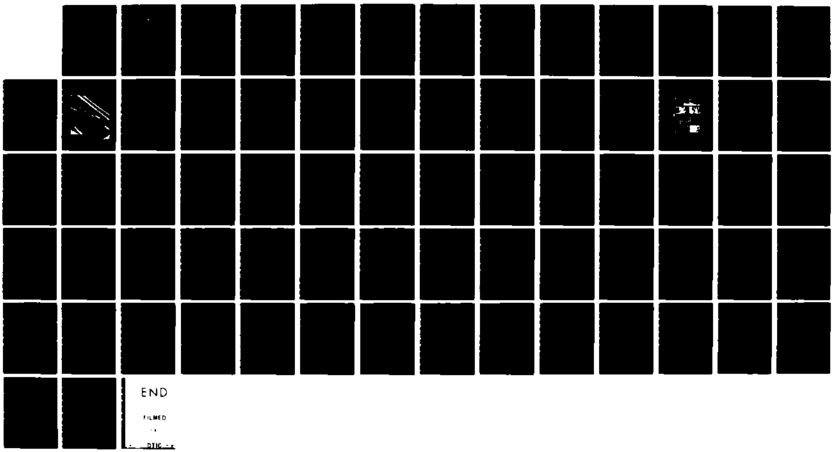

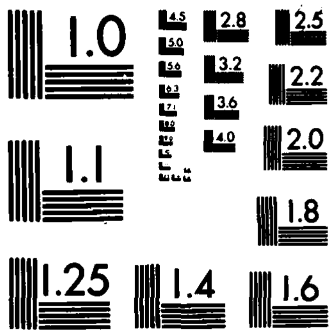

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METHODS OF IMPROVING THE MATRIX DOMINATED PERFORMANCE OF COMPOSITE STRUCTURES: A TECHNICAL REVIEW

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JULY 1983

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Contained herein is a discussion of methods of increasing the matrix dominated properties of two-phase, continuous fiber/polymer composite materials by the addition of a third phase. For example, a resultant composite exhibiting improved matrix dominated properties may consist of an epoxy matrix binding together both silicon carbide whiskers and continuous graphite fibers.

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The potential exists to hold the fiber volume content at a level of approximately 50 to 60 percent while displacing 5 to 15 percent of the epoxy with whiskers. The resultant fiber/whisker/epoxy composite has the potential to exhibit superior strength and stiffness to weight properties as compared to a fiber/epoxy composite.

Two distinct approaches are examined. The first approach utilizes the fibers as a carrier for the whiskers, or third phase. It is proposed that silicon carbide whiskers be grown directly on the surface of graphite fibers. These fibers can then be impregnated with the epoxy matrix. The second approach is to utilize the epoxy matrix as a carrier for the whiskers. Silicon carbide whiskers can be added to the epoxy prior to impregnation of the graphite fibers.

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SECTION 1.0
INTRODUCTION AND SUMMARY

The advantages of two-phase, composite materials, such as graphite/epoxy used in aircraft applications have been well documented. The main advantages of these materials over conventional metals can best be described in terms of superior strength and stiffness to weight ratios. In recent years the need to reduce fuel and strategic material usage while increasing payload and range have led to significant two-phase, composite material applications on commercial and military aircraft. The U.S. Navy, in particular, has been forthright and a leader in the application of these materials to operational aircraft structures, as evidenced by the F-14, F-18, and AV-8B aircraft.

It has been shown, both theoretically and experimentally, that the strength and stiffness of a given material is highly geometry dependent. For example, superior properties can be obtained from a material in the fibrous form as compared to the bulk form. Carrying this geometric process one step further, even higher properties can be obtained if the material is examined in the form of whiskers.

Material usage in the whisker form has not achieved the same level of success as fibrous material. The major emphasis and success of whisker research has been to incorporate whiskers in a metal matrix. The resultant composite exhibits superior properties as compared to the metal alone and provides a mechanism to extract to some extent the superior

properties of the whiskers. Whiskers have been incorporated in polymeric matrices, but these composites have not demonstrated their ability to outperform fiber/polymer composites.

Contained herein is a discussion of methods of increasing the matrix-dominated properties of a two-phase, fibrous, composite material by the addition of a third phase. These three-phase materials will consist of an epoxy matrix which binds together both fibrous and whisker reinforcements. The potential exists to hold the fiber volume content at a level of approximately 60 percent while displacing a percentage (5 to 15 percent) of the epoxy with whiskers. The resultant fiber/whisker/epoxy composite material has the potential to increase the strength- and stiffness-to-weight properties by 20% as compared to a fiber/epoxy composite. Although this potential has not been experimentally verified for polymeric matrix materials, it has been verified for aluminum matrix composites containing both silicon carbide whiskers and continuous filaments.

Two distinct approaches can be taken. The first approach utilizes the fibers as a carrier for the whisker, or third phase. Silicon carbide whiskers can be grown directly on the surface of graphite fibers. These fibers can then be impregnated with the epoxy matrix. The second approach is to utilize the epoxy matrix as a carrier for the whiskers. Silicon carbide whiskers can be added to the matrix prior to impregnation of the graphite fibers.

A review of pertinent experimental and theoretical studies has been conducted to support the potential of these two approaches. Estimated mechanical property improvements are presented along with a discussion of their theoretical basis. Recommendations are put forth on potential technical approaches to the two methods discussed.

SECTION 2.0

BACKGROUND

This section is not intended to represent a complete review of the available literature on the subjects to be discussed. Sufficient papers are reviewed to provide background support for the methods of improving matrix-dominated, composite material properties to be discussed.

2.1 Whiskered Fibers

During the late 1960's, poor fiber-to-matrix adhesion was a problem of major concern in the field of composite materials. Without the ability to consistently attain adequate fiber-to-matrix adhesion, the benefits of composite materials over conventional metals could not be realized. In 1967 the concept of whiskered fibers was developed by J. V. Milewski of Thermokinetic Fibers, Inc. and S. P. Prosen of the Naval Ordnance Laboratory (NOL) to promote better fiber-to-matrix adhesion (Reference 1). Whiskers grown at right angles to the fiber surface provided a means of mechanically anchoring the fiber in the matrix and thus resulted in good fiber-to-matrix "adhesion."

A total of five major contracts were issued by NOL over a four-year period to develop and evaluate this concept. In addition, the U. S. Army and U. S. Air Force each issued a contract to examine this concept. Numerous publications resulted from this work (References 1-13). During this period, however, other fiber surface treatment methods for promoting

proper fiber-to-matrix adhesion were developed, and further development of the whiskered fiber concept was terminated. The remainder of this discussion is based on selected results presented in References 1 through 13, and specific reference delineation is eliminated.

Whiskerizing was accomplished in both a batch and continuous mode of operation. The continuous mode setup is illustrated in Figure 1. Carbon monoxide (CO), methane (CH_4), and hydrogen (H_2) gases are fed into an electrically heated furnace which is at an optimum temperature of 1480°C. A ceramic brick of silicon dioxide (SiO_2) and carbon surrounds the fiber which is to be whiskered. The reaction of the vapor phase materials in the furnace causes crystals of β-silicon carbide (β-SiC) to nucleate on the surface of the fiber (see Figure 2). One of several vapor phase reactions which produces silicon carbide whiskers is:

$$SiO_2 + C \rightarrow SiO + CO$$

$$SiO + CH_4 \rightarrow SiC + H_2O + H_2$$

By controlling the temperature, gas feed rates and fiber feed rate, the diameter, length, and population of the whiskers on the fiber surface can be controlled. Whisker diameters and lengths varied from 100 Å to 0.5 μ and 0.1 to 5 μ, respectively. Whiskers were found to grow around each fiber in a fiber bundle for the continuous process and around each fiber in a dry layup for the batch (noncontinuous) process.

G.T.C. WHISKERIZING APPARATUS

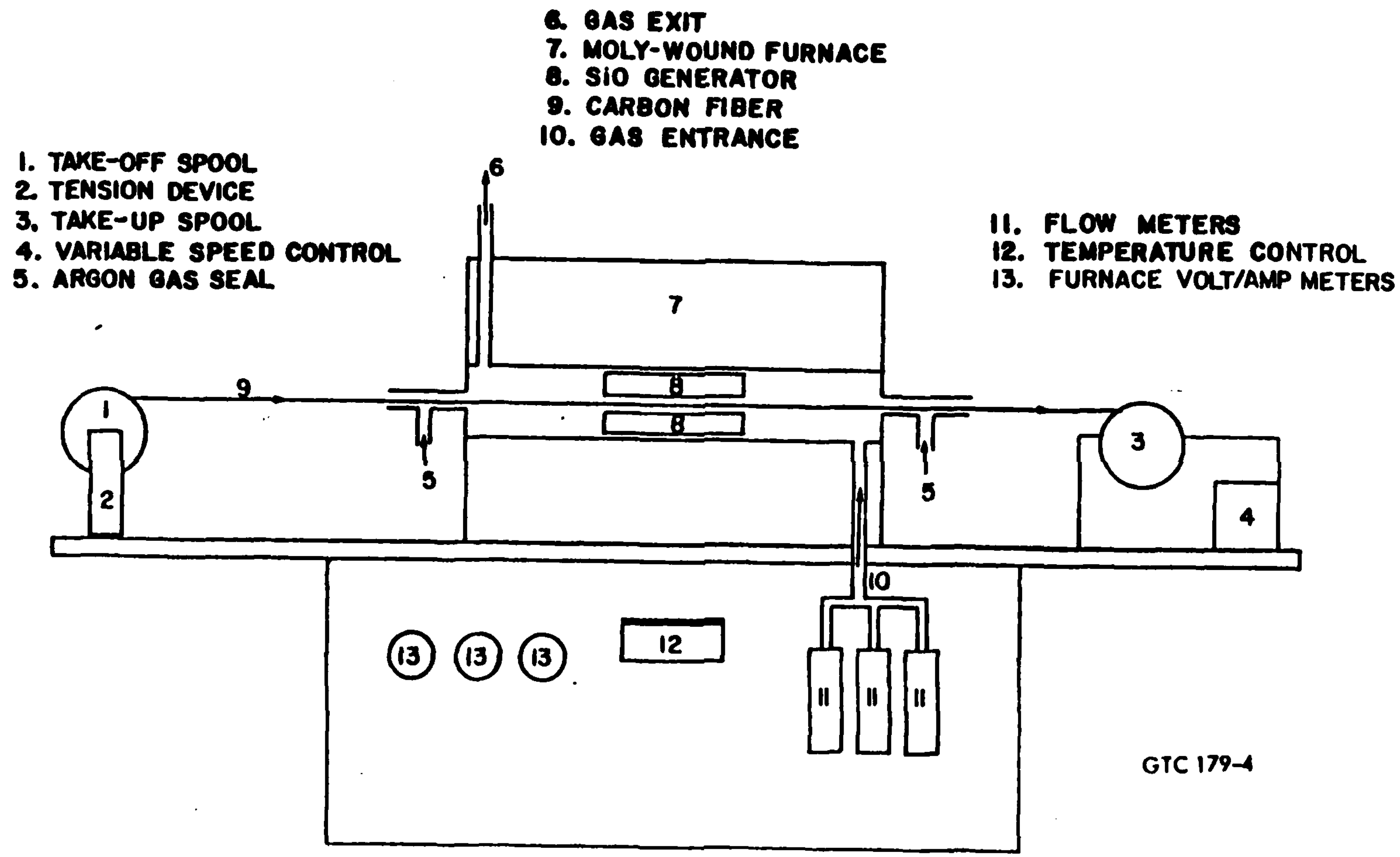


FIGURE 1. SCHEMATIC OF GENERAL TECHNOLOGIES CORPORATION'S CONTINUOUS WHISKERING APPARATUS (ADAPTED FROM REFERENCE 3).

"WHISKERIZED" R.A.E. BARBED WIRE TREATMENT

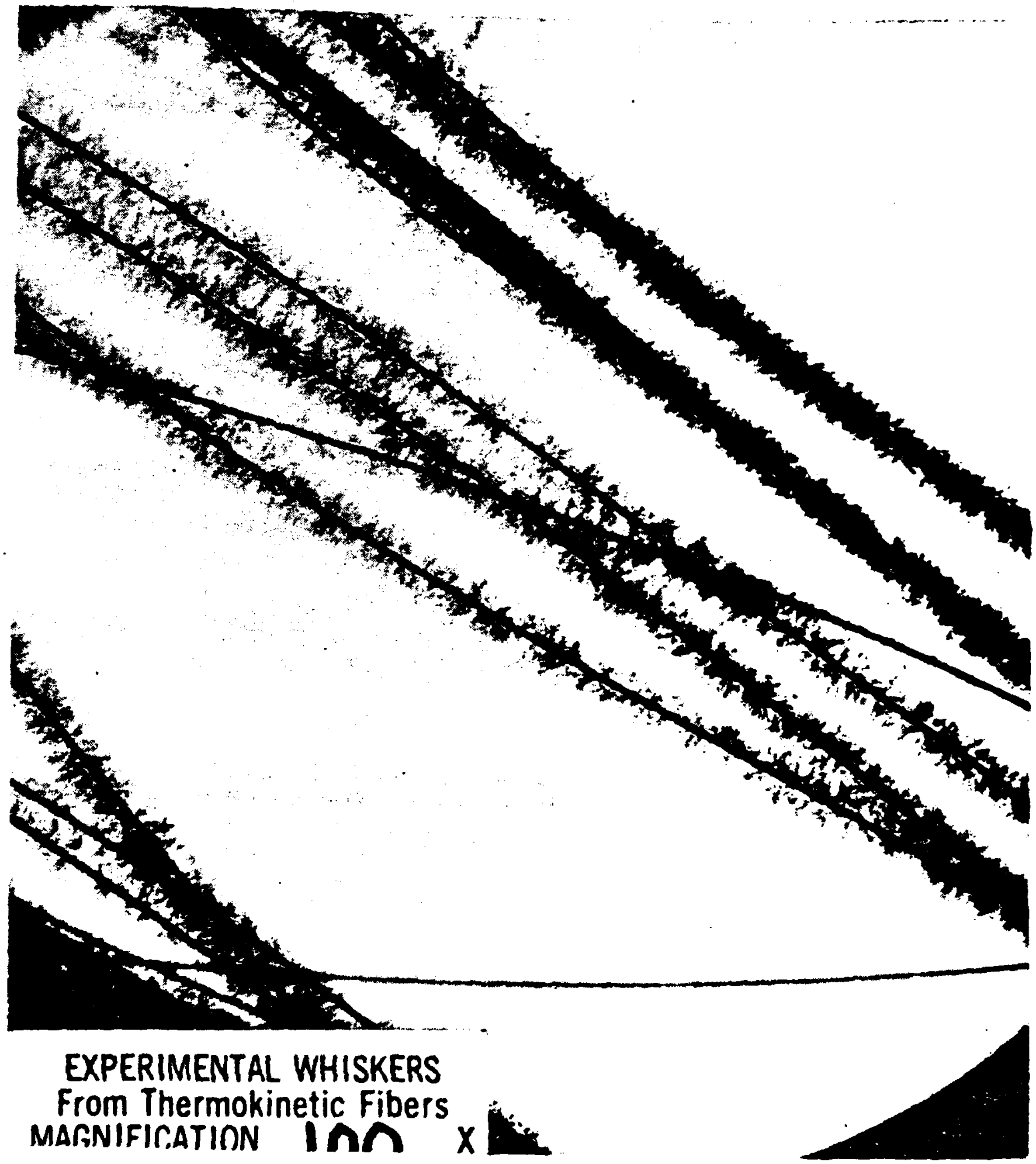


FIGURE 2. GRAPHITE FIBERS WHISKERIZED WITH SILICON CARBIDE (COURTESY OF J.V. MILEWSKI).

The batch and continuous processes can be used to successfully grow whiskers on any fiber or bulk material which can sustain the cited furnace temperature. The studies discussed here focussed on growing silicon carbide whiskers on various commercially available graphite fibers. The whiskered fibers were then impregnated with various commercially available epoxies to produce specimens from which mechanical properties could be measured.

Of the two process modes examined, batch and continuous, the continuous mode was considered optimum. The selected mechanical properties discussed below were determined from unidirectional composites composed of epoxy with whiskered and unwhiskered graphite fibers. Whiskered fibers were produced by the continuous process with the resulting composite compared directly to the unwhiskered fiber composite.

The data generated from the whiskered-graphite/epoxy specimens showed significant scatter. This scatter is attributed to two factors: 1) At the time this work was conducted, graphite fibers were relatively new, resulting in variable properties from batch-to-batch, and; 2) the whiskering process was also new and variable. Whiskering was found to have the following effects on mechanical properties:

- Fiber tensile strength degraded from 0 to 70 percent (15 percent nominally). The percent decrease in fiber tensile strength was partially dependent on the fiber supplier.

- Unidirectional compressive strength (0° direction) of the whiskered fiber composite varied from -66 to +2 percent as compared to the unwhiskered fiber composite.

- Composite flexure strengths (0° direction) were reduced by 5 to 15 percent. Unwhiskered specimens were found to fail by buckling or interlaminar shear failure. Whiskered fiber specimens were found to fail in a tensile mode with a resulting crack propagation.

- Interlaminar shear strength was found to increase by 300 percent on the average. However, the whiskered short beam shear specimens failed in flexure, not shear. Whiskering raised the interlaminar shear strength to the value typically found for current graphite/epoxy composites. It should be noted that the graphite fibers currently used are surface treated to promote proper fiber-to-matrix adhesion. The unwhiskered graphite fibers used for the work being discussed had no such treatment.

- Transverse (90° direction) tensile, compressive, and flexural strengths were all found to increase by 200 to 300 percent. All these transverse strengths increased with increasing whiskering up to a value of 5 percent whisker content. Above 5 percent whisker content, these strengths began to decrease from the peak values.

- Torsional fatigue was examined using NOL rings. The stiffness deterioration rate during cycling was found to be the same for whiskered and unwhiskered specimens. Whiskered ring specimens were found to withstand greater twist angles without breaking.

- Izod impact tests indicated no impact strength difference between whiskered and unwhiskered specimens. The failure mode for whiskered specimens was tensile, while the unwhiskered specimens exhibited interlaminar shear failure.

- The whiskering process was not found to affect moduli.

To some extent, the variability of the whiskerizing process and the varying quality of the fibers as supplied by the manufacturers contributed to the mixed results and data scatter discussed above. Fiber degradation due to whiskering and porosity due to poor specimen fabrication were major additional problems.

As delineated earlier, water vapor is produced as a by-product of the whiskering process. Excessive moisture erodes the graphite fiber by the following process:

$$C + H_2O \rightarrow CO + H_2$$

Fiber erosion resulted in strength reductions for fiber-dominated properties. However, there were instances where fiber-dominated properties were not degraded. This indicates the possibility to control water-vapor, fiber erosion and thus eliminate decreases in fiber-dominated properties.

Matrix-dominated properties were increased on average. Increases are attributed to whisker reinforcement of the matrix. Specimen void content varied from 0 to 5 percent by volume and this is partially responsible for the large scatter in data. The void content problem encountered was probably due to the laminate fabrication process using a press instead

of an autoclave. Removal of trapped gases is more difficult to achieve in a press as compared to an autoclave.

Recently, a contract was issued by Naval Sea Systems Command to produce whiskerized graphite fibers for incorporation in metal matrix composites. Contract N00024-81-C-5367 was issued to Versar, Inc., during September, 1981. Thermokinetic Fibers, Inc. and General Technologies Corporation, the original graphite whiskering developers, are now components of Versar, Inc. Contract progress reports (References 14-17) to date, indicate that the whiskering process has reduced fiber strengths. The fibers have been whiskered using the same process developed over a decade ago without any process modifications to control fiber erosion. An improved whiskering process which eliminates or minimizes fiber erosion has yet to be developed. A final report on this contract is not available at this time.

2.2 Whiskers, Particulates, and Microfibers

Whiskers, particulates, and microfibers are three distinct categories of noncontinuous material which are available for supplemental reinforcement in a continuous-fiber/polymer composite material. A whisker is essentially a perfect single crystal fiber having an aspect ratio (length-to-diameter ratio) of 10 to 100. Whisker diameters range from submicron to several microns. Microfibers are either polycrystalline fibers or imperfect single crystal fibers. Microfiber diameters and aspect ratios are the same as those found in whiskers. For the purposes of this discussion, a particulate will be considered as any material, crystalline or amorphous, with an aspect ratio less than 10.

The use of whiskers has lagged the use of continuous fibers in aerospace grade composite materials (References 18-21). The most successful application of whiskers has been to provide reinforcement in metal matrix composites (References 22, 23). Whiskers having aspect ratios less than 10, and therefore classified as particulates, have also been successfully applied in metal matrix composites (Reference 24). Whiskers have rarely been used in polymer composites due to less expensive particulate alternatives.

A search of the literature has revealed four types of microfibers (Reference 25). Of these four, two are no longer commercially available, and the remaining two are available from pilot-plants only. Due to the questionable continued availability of these microfibers, they should not be considered for application.

As mentioned, particulates have been used in aerospace grade metal matrix composite materials. Particulates have also been extensively used in polymer composites of below aerospace grade (References 25-28). In non-aerospace applications, particulates are generally termed as fillers. The term filler connotes a material which is used to take up space. In fact, these particulates or fillers are used to displace the plastic which is the more expensive constituent. These particulates, besides reducing final product cost, have the added benefit of improving mechanical properties. The property improvements, although significant in some instances, are not sufficient to permit use in demanding aerospace applications.

A search of the literature has not revealed any instance where whiskers, microfibers, or particulates have been used to provide supplementary reinforcement (as a third phase) in continuous-fiber/polymer composites.

A survey is currently being conducted by the author to determine the most promising whiskers and particulates available for use as a third-phase reinforcement. Potential third-phase reinforcement materials should meet the following criteria:

1) The third phase shall not be classified as a strategic material as defined by Public Law 96-41, "The Strategic And Critical Materials Stock Piling Revision Act Of 1979." (Reference 29).

2) The third phase shall be commercially available with good prospects for continued availability.

3) The third phase shall possess a diameter and aspect ratio which will not disrupt or interfere with the packing density of the continuous-fiber phase (See Section 2.3).

2.3 Packaging Concepts

The microstructural arrangement of a typical, two-phase, graphite/epoxy composite material system is illustrated in Figure 3. From a manufacturing and material property standpoint, the optimum fiber volume is approximately 60 percent. Regions are found where the fibers are arranged in hexagonal, square, and random arrays. The regions where hexagonal and square arrays exist have fiber volumes of approximately 90 and 78 percent, respectively. Regions where random arrays exist have fiber volumes of less than 78 percent.

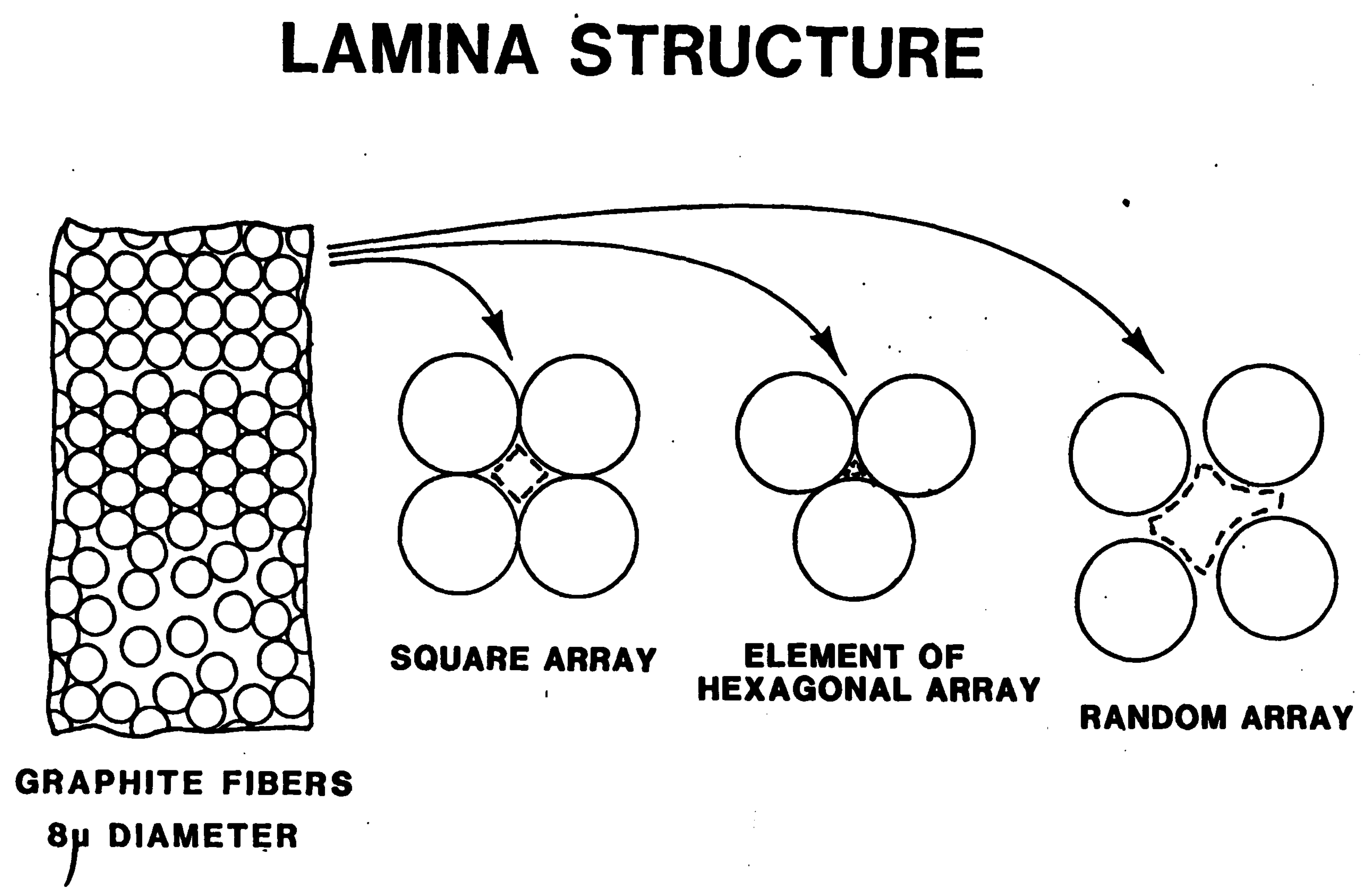


FIGURE 3. MICROSTRUCTURAL ARRANGEMENT OF A TYPICAL GRAPHITE/EPOXY COMPOSITE MATERIAL.

Consider the addition of a spherical particulate (aspect ratio = 1) to the epoxy matrix. The particulate has to be of the proper size so as not to disrupt fiber packing. Examination of the hexagonal array for 8 μ diameter fibers indicates that the maximum particulate diameter which can be accommodated without array disruption is approximately 1.2μ in diameter.

By adding 1.2μ diameter spherical particulates, the epoxy matrix has been transformed into a particulate/epoxy composite material matrix. Since the strength and stiffness of the particulate are greater than the strength and stiffness of the epoxy, the mechanical properties of the particulate/epoxy matrix will be superior to the mechanical properties of the epoxy matrix alone. Thus the resultant, three-phase, graphite/particulate/epoxy composite can be expected to have superior matrix-dominated properties when compared to the original, two-phase, graphite/epoxy composite. Although these property improvements have not been experimentally verified for polymeric matrix materials, it has been verified for aluminum matrix composites containing both silicon carbide and continuous filaments. These experimental results are discussed in Section 3.0.

If the particulate which is to be added possesses an aspect ratio greater than 1, then the diameter of that particulate must be less than 1.2μ, so as not to disrupt fiber packing. In addition, as the aspect ratio is increased, the reinforcement efficiency of the particulate increases (Reference 26) (see Section 2.5).

The gains in mechanical properties as a result of adding particulates or whiskers can be viewed in terms of the increased packing efficiency of the higher strength and stiffness constituents of the composite. An examination of the literature revealed that detailed studies of the packing concept discussed above have already been conducted by J. V. Milewski (References 30-34).

The packing concept is clearly illustrated in Figure 4. Two cylinders are partially filled with rods having an aspect ratio of 15. Spheres are introduced to each cylinder. In one case, the ratio of sphere-to-rod diameter, R, is 4, while in the other case R is 0.5. After mixing, the cylinder having R = 4 is found to have a void content of 50 percent, while the cylinder having R = 0.5 is found to have a void content of 35 percent. As R is decreased below 0.5, void content will also decrease. Note that in an actual composite of this form, the void content would translate into the matrix content.

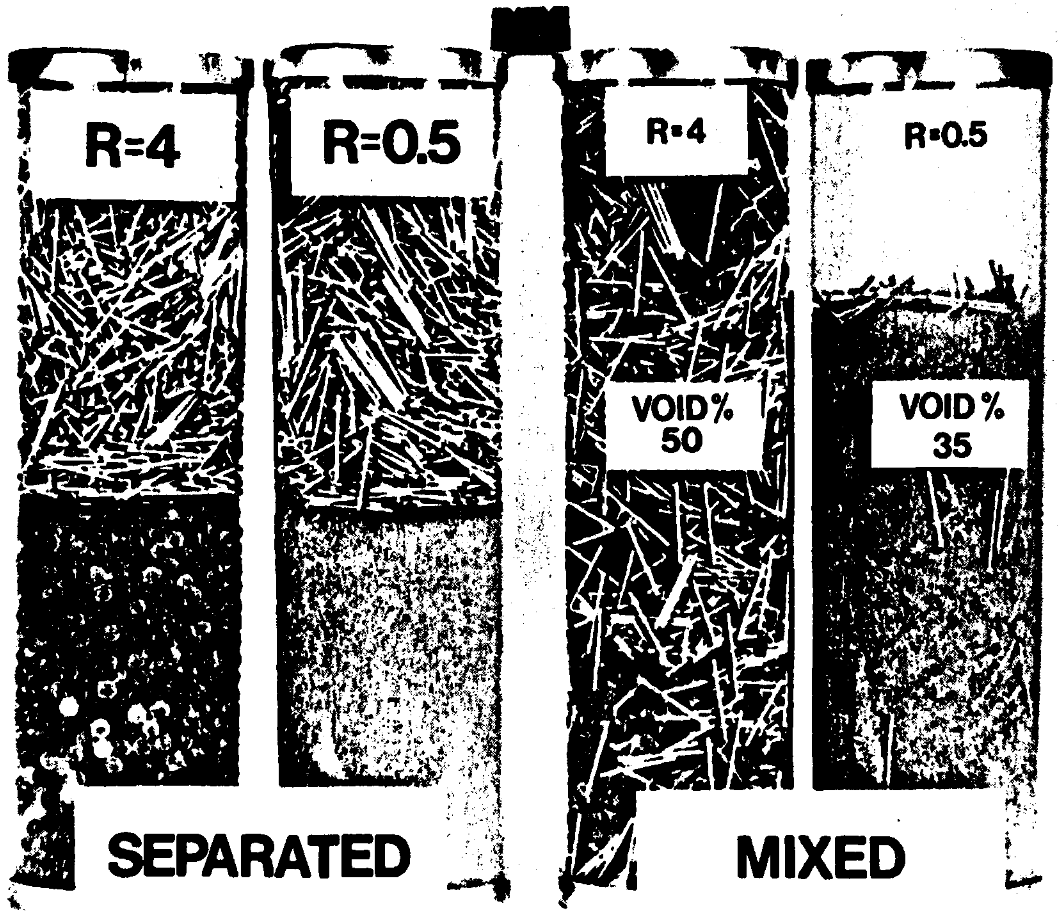
The third-phase particulate or whisker reinforcement is size critical (1.2μ maximum) and is dependent on the continuous-fiber diameter (8μ).

2.4 Epoxy Resins

The addition of particulates or whiskers to liquids may rheologically affect the liquid mixture by increasing viscosity and thixotropy (References 35, 36, and 37). The effect of particulate addition to a single component liquid system can readily be studied and is a function of the two components. If the liquid has two components, the determination of the cause/effect relationship between the liquid and particulate is a function of the three constituents.

FIGURE 4. EFFECT OF PARTICLE SIZE ON PACKING EFFICIENCY.

ALL CYLINDERS CONTAIN THE SAME VOLUME OF SOLIDS 3 PARTS SPHERES AND I PART FIBERS AT 15/1 L/D



(COURTESY OF J.V. MILEWSKI)

Consider a two-component resin system, such as Diglycidyl Ether of Bisphenol A (DGEBA) epoxide and Diethylenetriamine (DETA) curing agent. The addition of 2 to 6 percent by weight of fumed silica (particulate of 0.015 μ diameter) to this resin will transform it from a liquid to a gel (References 38, 39). It may be necessary to add solvents or diluents to the gel to obtain the flow characteristics required for adequate fiber impregnation and/or to increase the percentage of fumed silica desired. The percentage addition of solvent or diluent will not only affect processibility but will also affect cure and mechanical properties. The optimum system from a processibility/cure/mechanical properties standpoint will be a function of the four components and thus require careful study.

Now consider the 3501-6* hot-melt epoxy resin system currently being used in combination with graphite fibers to produce components for the F-18 and AV-8B aircraft. This resin consists of three different epoxides, Diaminodiphenylsulphone (DDS) curing agent, and Boron Trifloride (BF_3) catalyst (Reference 40). The addition of particulates or whiskers will increase viscosity and thixotropy. However, the optimum system is now a function of the original resin, the particulate or whisker, and one or more diluents or solvents. At a minimum, the optimum system will be a function of seven components** and will necessarily be complicated. A prudent approach to the matrix carrier method of third-phase reinforcement would be to conduct initial studies with a simple DGEBA/DETA type epoxy resin. Once the proof of concept has been demonstrated with a simple resin, additional studies of the more complex 3501-6 resin can follow.

* 3501-6 is a product of Hercules, Inc.
** Note that temperature, order of mixing, etc., have not been considered.

The silicon carbide whiskers are ridigly attached to the graphite fibers in the fiber carrier method of third-phase reinforcement. In this case, no rhelogical change is expected in the resin, and 3501-6 may be used.

2.5 Micromechanical Considerations

STIFFNESSES

Many approaches have been used to determine the stiffnesses of composite materials based on the stiffnesses of the constituent materials. Chamis and Sendeckyj (Reference 41) have critiqued theories predicting the thermoelastic properties of composite materials and have classified these theories as follows: netting analysis, mechanics of materials, self-consistent model, variational, exact (within the context of classical elasticity), statistical discrete elements, semiempirical methods, and theories accounting for microstructure.

Several basic assumptions common to all these theories are cited by Chamis and Sendeckyj, namely: 1) The ply is macroscopically homogeneous, linearly elastic, and generally orthotropic. 2) The fibers are linearly elastic and homogeneous. 3) The matrix is linearly elastic and homogeneous. 4) Fiber and matrix are free of voids. 5) There is complete bonding at the interface of the constituents, and there is no transitional region between them. 6) The ply is initially in a stress-free state. 7) The fibers are a) regularly spaced, and b) aligned. These assumptions tend to unrealistically represent the physical state of the composite material. Due to these assumptions, precise prediction of composite stiffnesses is not possible, nor is it necessary from a practical standpoint. For design purposes, it is desirable to use a simple and rapid, approximate formulation to estimate composite properties based on constituent properties.

Two methods have been selected for stiffness estimates using the following formulations: a) The Halpin-Tsai equations (References 42-44),

$$E_1 \cong E_f V_f + E_m V_m \tag{2.1}$$

$$\nu_{12} = \nu_f V_f + \nu_m V_m \tag{2.2}$$

$$\frac{M}{M_m} = \frac{1 + \xi\eta V_f}{1 - \eta V_f} \tag{2.3}$$

$$\eta = \frac{(M_f/M_m) - 1}{(M_f/M_m) - \xi} \tag{2.4}$$

where M = Composite Modulus E_2 or G_{12}

M_f = Fiber Modulus E_f or G_f

M_m = Matrix Modulus E_m or G_m

ξ = Empirically determined measure of fiber reinforcement that depends on loading conditions, packing geometry, and fiber geometry. Suitable values of ξ are:

$\xi_{E_{12}}$ = 2, 2 (a/b)

$\xi_{G_{12}}$ = 1, $(1 + 40\ V_f^{10})$ and $\text{LOG}\ \xi_{G_{12}} = \sqrt{3}\ \text{LOG (a/b)}$

(a/b = Fiber Aspect Ratio)

and for random, discontinuous-fiber reinforcement,

$$E = 3/8\ E_1 + 5/8\ E_2 \tag{2.5}$$

and; b) the Paul equations (Reference 45),

$$\frac{E}{E_m} = \frac{E_m + (E_d - E_m)\, V_d^{2/3}}{E_m + (E_d - E_m)\, V_d^{2/3}\, (1-V_d^{1/3})} \tag{2.6}$$

$$\frac{G}{G_m} = \frac{G_m + (G_d - G_m)\, V_d^{2/3}}{G_m + (G_d - G_m)\, V_d^{2/3}\, (1-V_d^{1/3})} \tag{2.7}$$

$$\nu = \left(\frac{E}{2G}\right) - 1 \tag{2.8}$$

where, subscript d specifies the dispersed reinforcement.

The first method will use the Halpin-Tsai Equations to predict the properties of the epoxy and whisker (or particulate) combination. These calculated properties will then be substituted back into the Halpin-Tsai equations along with graphite fiber properties to obtain the properties of the resultant three-phase composite material.

The second method will use the Paul equations to predict the properties of the epoxy and whisker (or particulate) combination. These calculated properties will then be substituted into the Halpin-Tsai equations along with graphite fiber properties to obtain the properties of the resultant three-phase composite material.

CRITICAL FIBER LENGTH

Consider a whisker or discontinuous fiber as illustrated in Figure 5. As the composite is loaded, stress is transferred from the matrix to the fiber via shear. At the end of the fiber, $x = 0$, the fiber stress is essentially zero, and the interfacial shear stress if finite. As x increases, the fiber stress increases up to the maximum fiber stress, $(\sigma_f)_{max}$, and the interfacial shear stress, τ, reduces to zero.

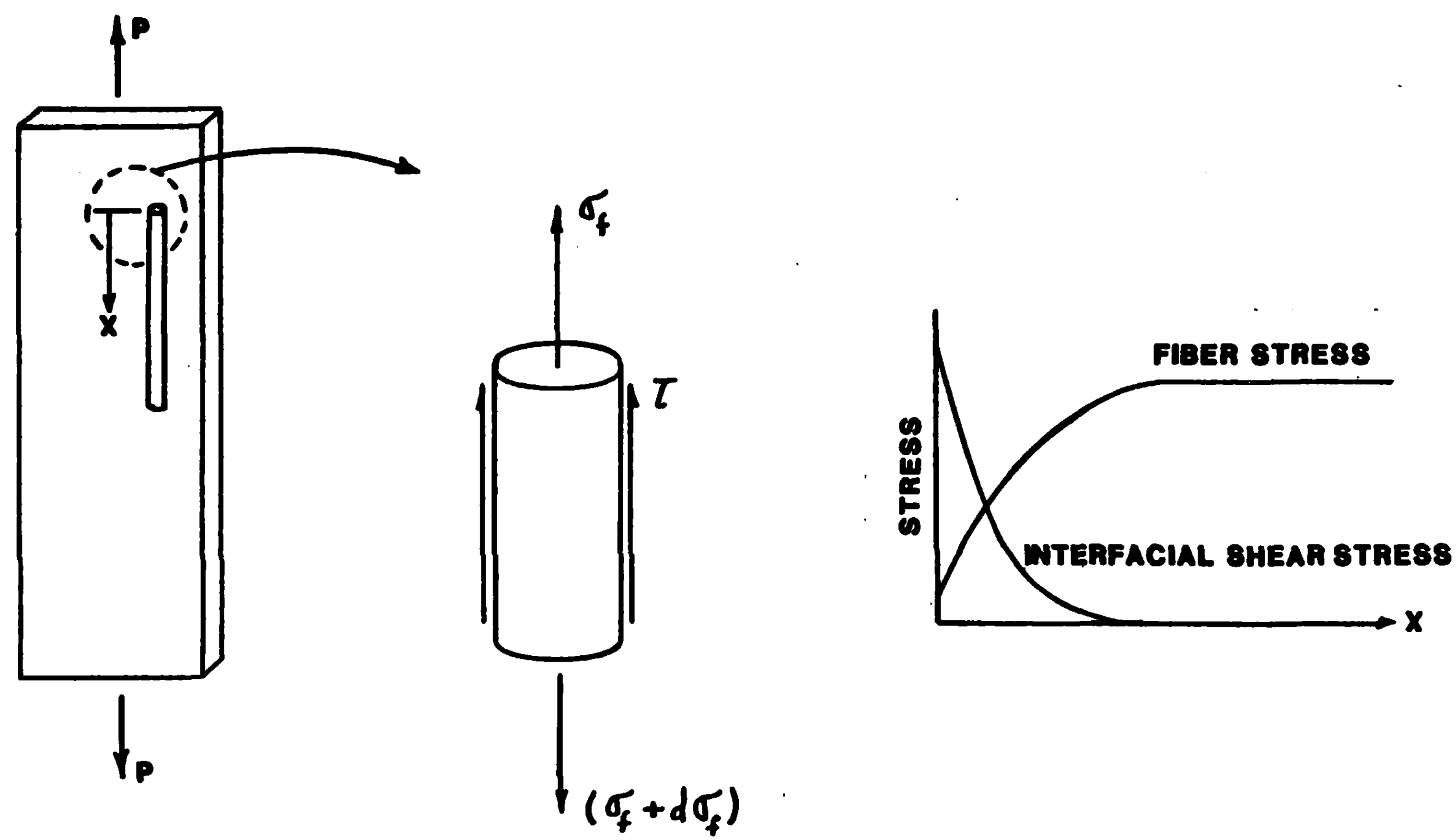


FIGURE 5. STRESS TRANSFER FROM MATRIX TO DISCONTINUOUS FIBER.

Therefore, to obtain the most efficient use of a discontinuous reinforcement, a critical fiber length, l_c, must be exceeded. The length of the fiber over which the fiber stress is not $(\sigma_f)_{max}$ is termed the ineffective length or critical fiber length, l_c (Reference 46). The determination of l_c is dependent upon the properties of the fiber and matrix and whether the matrix is considered elastic, plastic, or elastic-plastic. For this discussion, the exact equation required to determine l_c is not important. Various methods of determining l_c are presented in Reference 26.

If whiskers are added to a continuous-fiber, graphite/epoxy composite, then the whiskers should have a length greater than l_c in order to obtain the most efficient reinforcement possible. However, as the whisker length or aspect ratio increases, rheological problems may occur during manufacture and continuous fiber packing may be disrupted (see Sections 2.3 and 2.4). From a manufacturing and packing standpoint, it may not be possible to use whiskers or particulates whose length exceeds l_c. This fact should not be unsettling. Property improvements can be realized even though the <u>most</u> <u>efficient</u> use of the whiskers or particulates may not be realized.

STRENGTH IN GENERAL

Methods of determining the tensile, compressive, and shear strengths of composite materials based on constituent data have not received as extensive an examination as methods for determining stiffnesses.

The strengths of a unidirectional, continuous-fiber composite due to transverse tension, transverse compression, and inplane shear are matrix-dominated properties. Formulations which adequately predict these strengths have not been found in the literature, however, the modes of failure are known (Reference 46). Composite failure modes due to transverse tension are (1) constituent debonding and/or fiber splitting and (2) matrix tensile failure. Composite failure modes due to transverse compression are (1) matrix shear failure or (2) matrix shear failure with constituent debonding and/or fiber crushing. Composite failure modes due to inplane shear are (1) matrix shear failure, (2) constituent debonding, and (3) matrix shear failure with constituent debonding. The addition of whiskers or particulates which increase the effective E and G of the epoxy will lead to increases in transverse tension, transverse compression, and inplane shear strengths.

LONGITUDINAL TENSILE STRENGTH

The tensile strength of an aligned, continuous-fiber reinforced composite is given by Reference 26:

$$(\sigma_{tu})_{cf} = \sigma_{fu} V_f + (\sigma_m)_{\epsilon_f'} (1 - V_f) \quad , \text{ for } V_f \geq V_{min} \qquad (2.9)^*$$

where $(\sigma_{tu})_{cf}$ = ultimate tensile strength of continuous fiber composite

σ_{fu} = ultimate fiber tensile strength

$(\sigma_m)_{\epsilon_f'}$ = matrix stress when fibers are strained to their ultimate tensile strain

V_{min} = minimum fiber volume fraction necessary for fiber reinforcement

* non-statistical approach

The tensile strength of an aligned, discontinuous-fiber reinforced composite is given by Reference 26:

$$(\sigma_{tu})_{df} = \sigma_{fu} V_f \left[1-(1-\beta)\, l_c/l \right] + (\sigma_m)_{\epsilon_f}' (1-V_f) \quad , \text{ for } V_f > V_{min} \qquad (2.10)^*$$

where $(\sigma_{tu})_{df}$ = ultimate tensile strength of discontinuous fiber composite

l = length of discontinuous fiber

$\beta = \sigma_f/(\sigma_f)_{max}$ for $l < l_c/2$

A comparison of these two equations indicates that continuous fibers strengthen a composite to a higher degree than discontinuous fibers. The effect on the tensile strength of a continuous-fiber graphite/epoxy composite (V_f = .60) with a percentage of the matrix replaced with randomly oriented whiskers should be minimal. The longitudinal tensile strength of this type of composite will be dominated by the continuous fiber strength with little contribution from the whiskers.

LONGITUDINAL COMPRESSIVE STRENGTH

The compressive strength of an aligned continuous-fiber composite is governed by fiber buckling. Two modes of fiber buckling can occur and are illustrated in Figure 6. The fibers can buckle out-of-phase, termed the extensional mode, or in-phase, termed the shear mode. For fiber volume fractions less than .30, the extensional mode governs compressive strength (Reference 47). For practical fiber volume fractions, $V_f \geq .40$, the shear mode governs compressive strength (Reference 46). For the shear mode, the maximum composite compressive stress and corresponding critical

* non-statistical approach

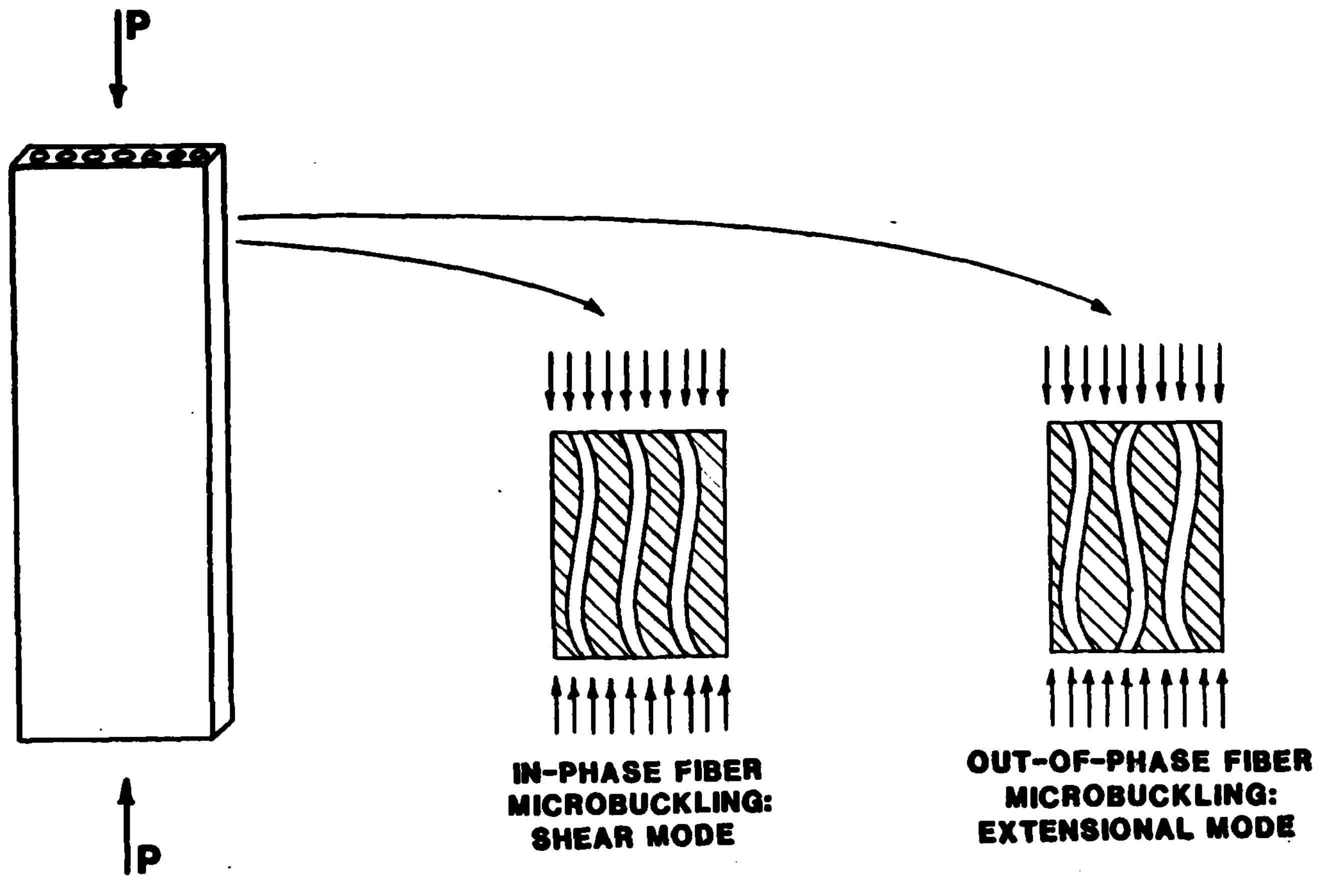


FIGURE 6. FIBER MICROBUCKLING MODES DUE TO LONGITUDINAL COMPRESSION.

strain are:

$$\sigma_{c_{max}} = \frac{G_m}{1-V_f} \tag{2.11}$$

$$\epsilon_{cr} = \frac{1}{V_f(1-V_f)} \left[\frac{G_m}{E_f}\right] \tag{2.12}$$

Equation 2.11 predicts a higher compressive strength than experimentally observed even if the matrix shear modulus is allowed to vary nonlinearly approximate inelastic behavior. As delineated in Reference 46 at $V_f \geq .40$, buckling may be preceded by matrix yield, matrix microcracking, and/or constituent debonding. The compressive failure may be initiated when the transverse tensile strain exceeds the composite transverse strain capability, resulting in cracks at the interface. Accounting for the failure initiating transverse strain, the maximum composite compressive stress becomes (Reference 46),

$$\sigma_{c_{max}} = \frac{(E_f V_f + E_m V_m)(1-V_f^{1/3})\, \epsilon_{mu}}{\nu_f V_f + \nu_m V_m} \tag{2.13}$$

where ϵ_{mu} = ultimate matrix strain

Equation 2.13 shows much better agreement with experimental data than Equation 2.11.

For aligned, discontinuous-fiber composites, the maximum compressive strength is dependent on the fiber length and is given by

$$\sigma_{c_{max}} = \left[\frac{\tau_y l}{d}\right] V_f + \sigma_{mu} V_m, \text{ for } l < l_c \qquad (2.14)$$

$$\sigma_{c_{max}} = \sigma_{fu} (1 - l_c/2l) V_f + (\sigma_m)_{\epsilon_f'} V_m, \text{ for } l > l_c \qquad (2.15)$$

$$\sigma_{c_{max}} = \sigma_{fu} V_f + (\sigma_m)_{\epsilon_f'} V_m, \text{ for } l \gg l_c \qquad (2.16)$$

where σ_{mu} = matrix ultimate stress

τ_y = interface shear stress

d = fiber diameter

Equations (2.14), (2.15), and (2.16) assume that the discontinuous-fiber volume fraction is above V_{min}.

Formulations for the determination of the compressive strength of a composite consisting of both continuous and discontinuous fibers have not been found in the literature.

Consider a unidirectional graphite/epoxy composite with V_f = .60. If some of the matrix is replaced with whiskers, the resultant matrix (a composite itself) will have higher stiffnesses than the original matrix alone. If G_m and E_m are greater for the epoxy/whisker matrix, then examination of Equations 2.11, 2.12, and 2.13 indicates that the longitudinal compressive strength of the composite will increase. The amount of strength improvement will be dependent on the whisker length and volume fraction. Foye (Reference 48), determined that the addition

of a 4 percent concentration of sapphire whiskers will raise the matrix shear modulus by 70 percent and will raise the resistance to compression crippling by 62 percent.

TRANSPORT PROPERTIES

Coefficients of thermal expansion, diffusion constants, and heat conduction (commonly referred to as transport properties), can also be predicted for a composite material based on a knowledge of the constituent materials (References 49, 50).

3.0 MATERIAL SYSTEM DESIGN

The methods presented in this section are applicable to both the fiber carrier and matrix carrier approaches to third-phase reinforcement*.

The addition of a third phase to a graphite/epoxy composite will result in a density increase. This can readily be seen by examining the density values given in Table 1. Fiber volume fractions of interest range from 50% to 65%. The increases in density caused by displacing a percentage of the epoxy matrix with silicon carbide whiskers for the volume fractions of interest are illustrated in Figure 7. This data has been replotted in Figure 8 to illustrate lines of 5, 10, 15, and 20 percent increases in density as functions of fiber volume fraction and percent displacement of the matrix. Figures 7 and 8 indicate that for a constant increase in density, a higher percentage of matrix can be displaced as the fiber volume fraction increases from 50% to 65%.

Using the Halpin-Tsai and Halpin-Tsai/Paul formulations described in Section 2.5, the stiffnesses of a composite consisting of various percentages of graphite fiber, epoxy matrix, and silicon carbide whiskers were calculated. The calculated stiffnesses using the Halpin-Tsai equations with whisker aspect ratios of 1 and 20 are given in Tables 2 and 3, respectively. As discussed in Section 2.5, more efficient reinforcement is achieved as the whisker aspect ratio increases. The Halpin-Tsai formulation is the only approximate method which can account for the aspect ratio of the reinforcement material. As indicated in Tables 2 and 3, as the whisker

*NOTE: The fiber carrier approach can only use silicon carbide as a third-phase reinforcement. The matrix carrier approach is not limited to the use of silicon carbide.

TABLE 1. MATERIAL PROPERTY DATA

Material	E (10^6 PSI)	G (10^6 PSI)	ρ (LBS/IN^3)
Graphite Fiber	34[1]	14*	.065[1]
Epoxy	.65[1]	.24[2]	.044[3]
Silicon Carbide	100[3]	38*	.115[3]

1 - From Reference 51

2 - From Reference 52

3 - From Reference 53

* - Estimated

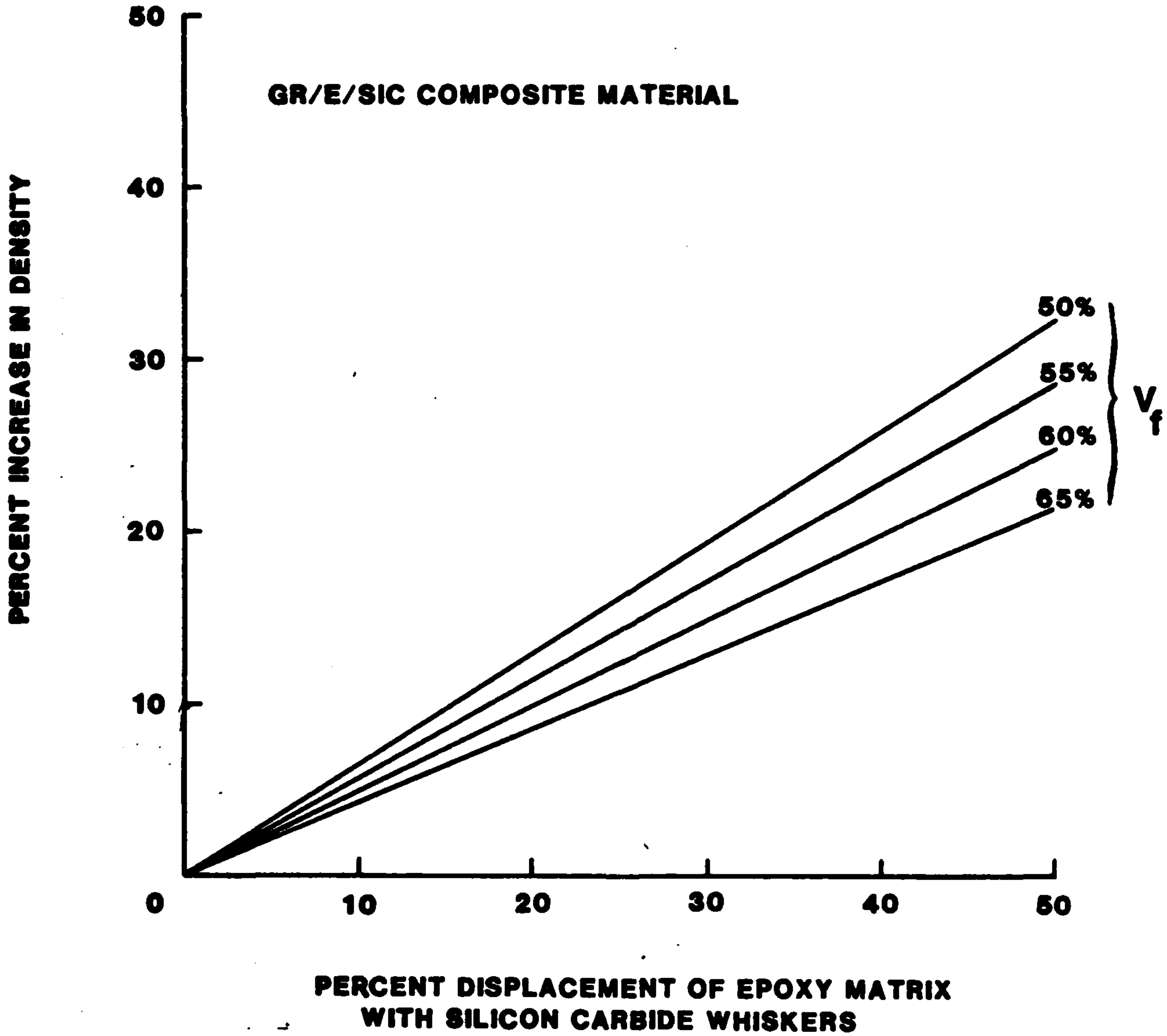


FIGURE 7. EFFECT ON COMPOSITE DENSITY DUE TO ADDITION OF SiC WHISKERS.

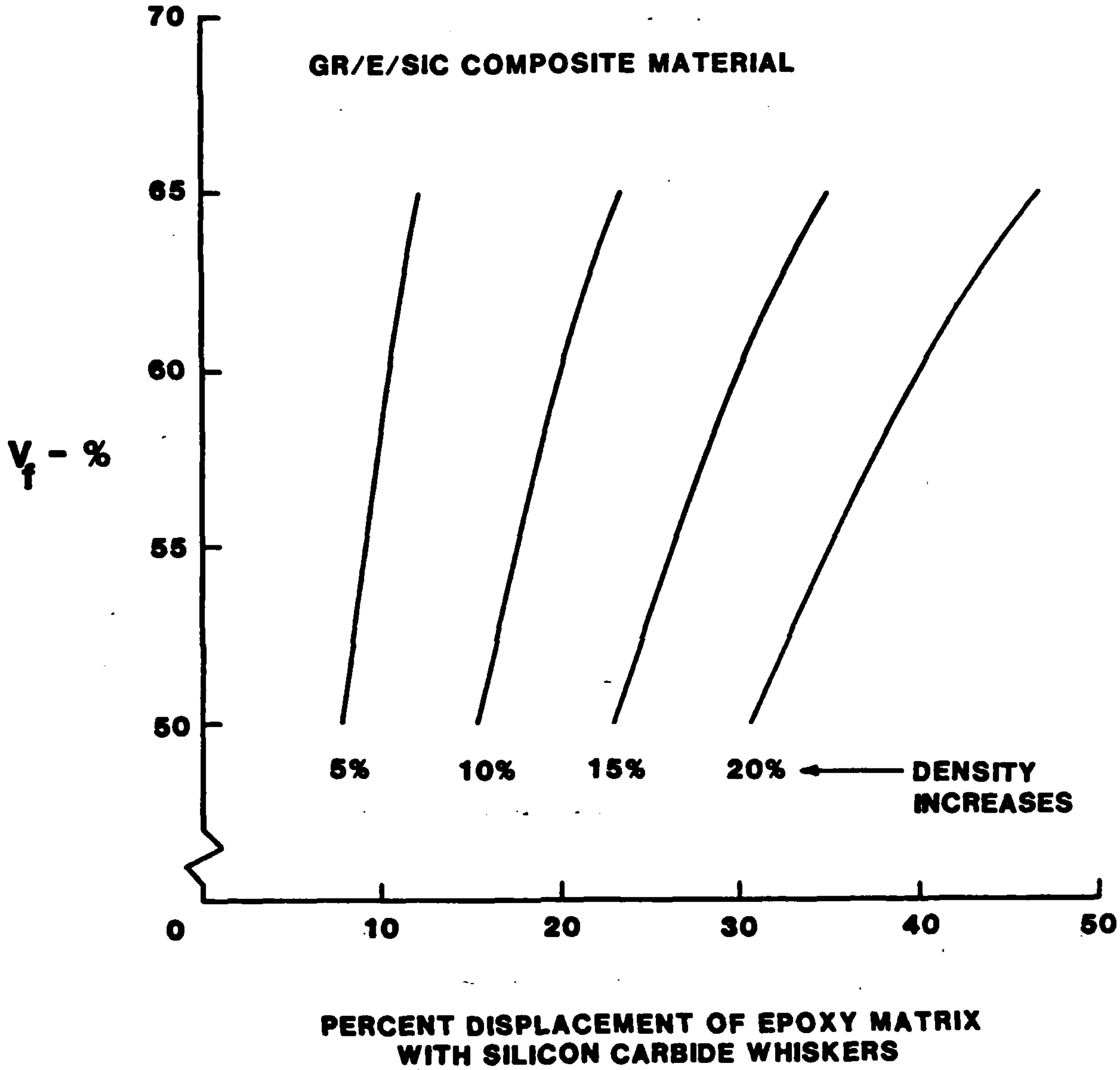


FIGURE 8. EFFECT ON COMPOSITE DENSITY DUE TO ADDITION OF SiC WHISKERS.

aspect ratio increases from 1 to 20, more efficient reinforcement is attained for a given whisker volume content. Calculated stiffnesses using the Halpin-Tsai/Paul equations are given in Table 4. For all the combinations listed in Tables 2, 3, and 4, Gr/E/SiC stiffness improvements are attained when compared to Gr/E values.

The stiffness data in Tables 2, 3, and 4 were combined with density data to examine specific stiffnesses. Specific stiffness data for Gr/E/Sic composites was then normalized with respect to Gr/E specific stiffness. Specific stiffness data for Gr/E/SiC composites was then normalized with respect to Gr/E specific stiffness. The resulting data, showing increases or decreases in specific stiffness as a function of matrix displacement with silicon carbide whiskers, is plotted in Figures 9, 10, and 11. An examination of these plots indicates that specific E_1 decreases, i.e., the addition of silicon carbide reduces material efficiency with respect to E_1. This result is not surprising since E_1 is a graphite fiber controlled property. The addition of silicon carbide results in specific increases in E_2 and G_{12}, i.e., as silicon carbide is added, E_2 and G_{12} increases offset density increases. The results for E_2 and G_{12} are as expected, since silicon carbide addition affects matrix dominated properties.

Improvements in stiffness and strength properties due to the addition of silicon carbide to a graphite/epoxy composite has not been verified experimentally. However, silicon carbide has been used to increase the transverse tensile strength and stiffness of a 6061 Aluminum/Borsic* composite (Reference 54). The major conclusions cited in Reference 54 are:

1) An increase in transverse tensile strength properties from 35 to 56 KPSI has been achieved by incorporation of 15% V_{SiC} whiskers in the

*Borsic filaments are silicon carbide coated boron filaments produced by Hamilton Standard, Division of United Aircraft Corporation.

TABLE 2. CALCULATED MODULI USING HALPIN-TSAI FORMULATION WITH WHISKER ASPECT RATIO EQUAL TO 1.

$V_f/V_E/V_{SiC}$	ρ (LBS/IN^3)	E_1 (10^6 PSI)	E_2 (10^6 PSI)	G_{12} (10^6 PSI)
50/50/0	.0545	17.32	1.27	.68
50/47.5/2.5	.0563	17.37	2.73	.75
50/45/5	.0580	17.43	3.09	.82
50/42.5/7.5	.0598	17.49	3.49	.91
50/37.5/12.5	.0633	17.64	4.39	1.10
50/32.5/17.5	.0669	17.83	5.48	1.35
50/25/25	.0722	18.26	7.63	1.87
65/35/0	.0576	22.32	1.79	1.04
65/33.25/1.75	.0588	22.36	4.25	1.14
65/31.5/3.5	.0601	22.40	4.78	1.25
65/29.75/5.25	.0614	22.44	5.35	1.37
65/26.25/8.75	.0639	22.54	6.62	1.65
65/22.75/12.25	.0663	22.68	8.11	2.00
65/17.5/17/5	.0701	22.98	10.85	2.71

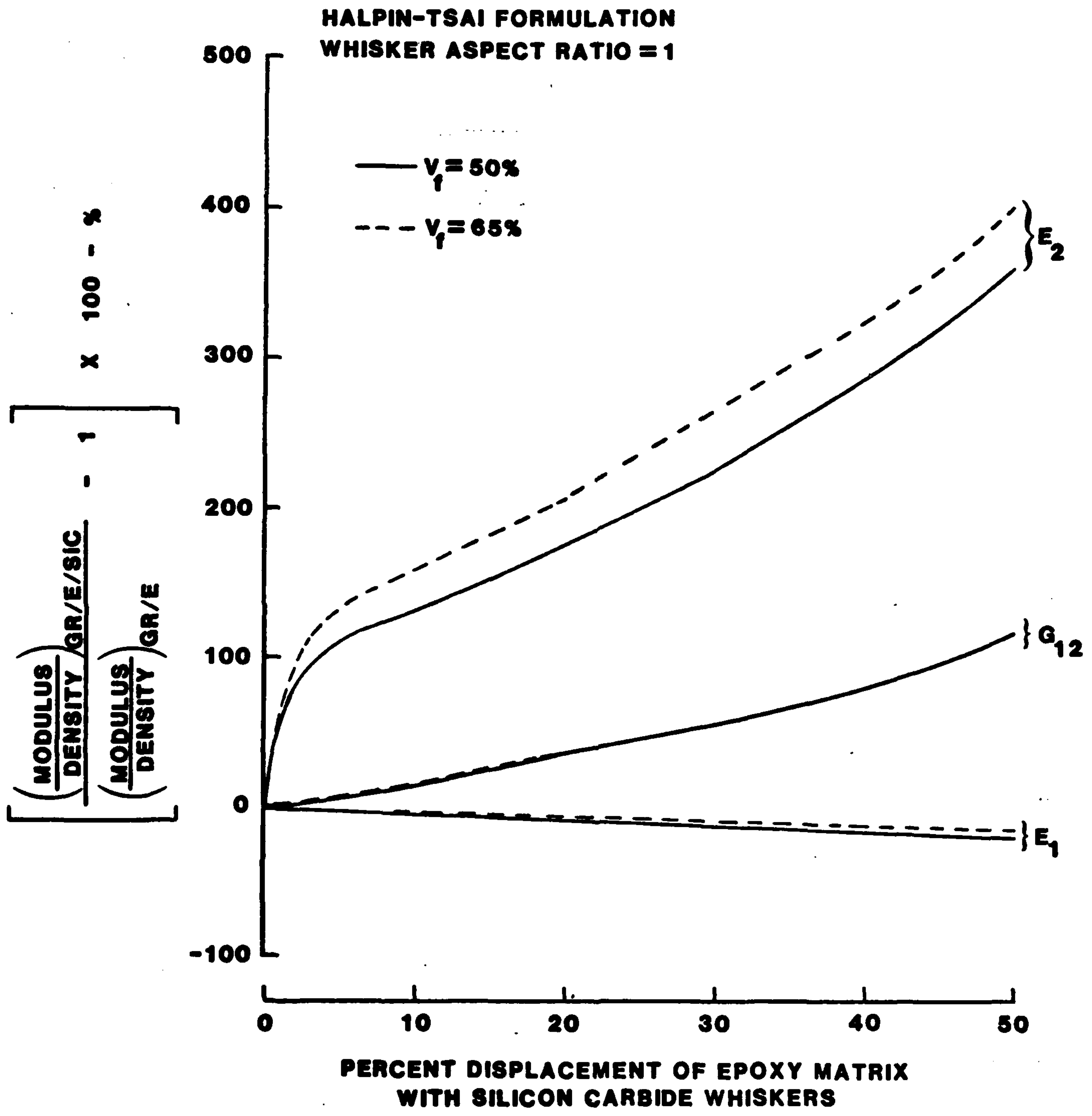


FIGURE 9. PROJECTED MODULUS IMPROVEMENTS DUE TO THE ADDITION OF SIC WHISKERS: HALPIN-TSAI FORMULATION WITH WHISKER ASPECT RATIO EQUAL TO 1.

TABLE 3. CALCULATED MODULI USING HALPIN-TSAI FORMULATION WITH WHISKER ASPECT RATIO EQUAL TO 20.

$V_f/V_E/V_{SiC}$	ρ (LBS/IN^3)	E_1 (10^6 PSI)	E_2 (10^6 PSI)	G_{12} (10^6 PSI)
50/50/0	.0545	17.32	1.27	.68
50/47.5/2.5	.0563	17.56	3.90	.76
50/45/5	.0580	17.82	4.37	.83
50/42.5/7.5	.0598	18.09	6.85	.88
50/37.5/12/5	.0633	18.75	9.70	1.11
50/32.5/17.5	.0669	19.54	12.50	1.36
50/25/25	.0722	21.15	16.77	1.91
65/35/0	.0576	22.32	1.79	1.04
65/33.25/1.75	.0588	22.49	5.94	1.35
65/31.5/3.5	.0601	22.67	8.01	1.48
65/29.75/5.25	.0614	22.86	9.88	1.55
65/26.25/8.75	.0639	23.32	13.31	1.94
65/22.75/12.25	.0663	23.87	16.38	2.32
65/17.5/17.5	.0701	25.00	20.57	3.13

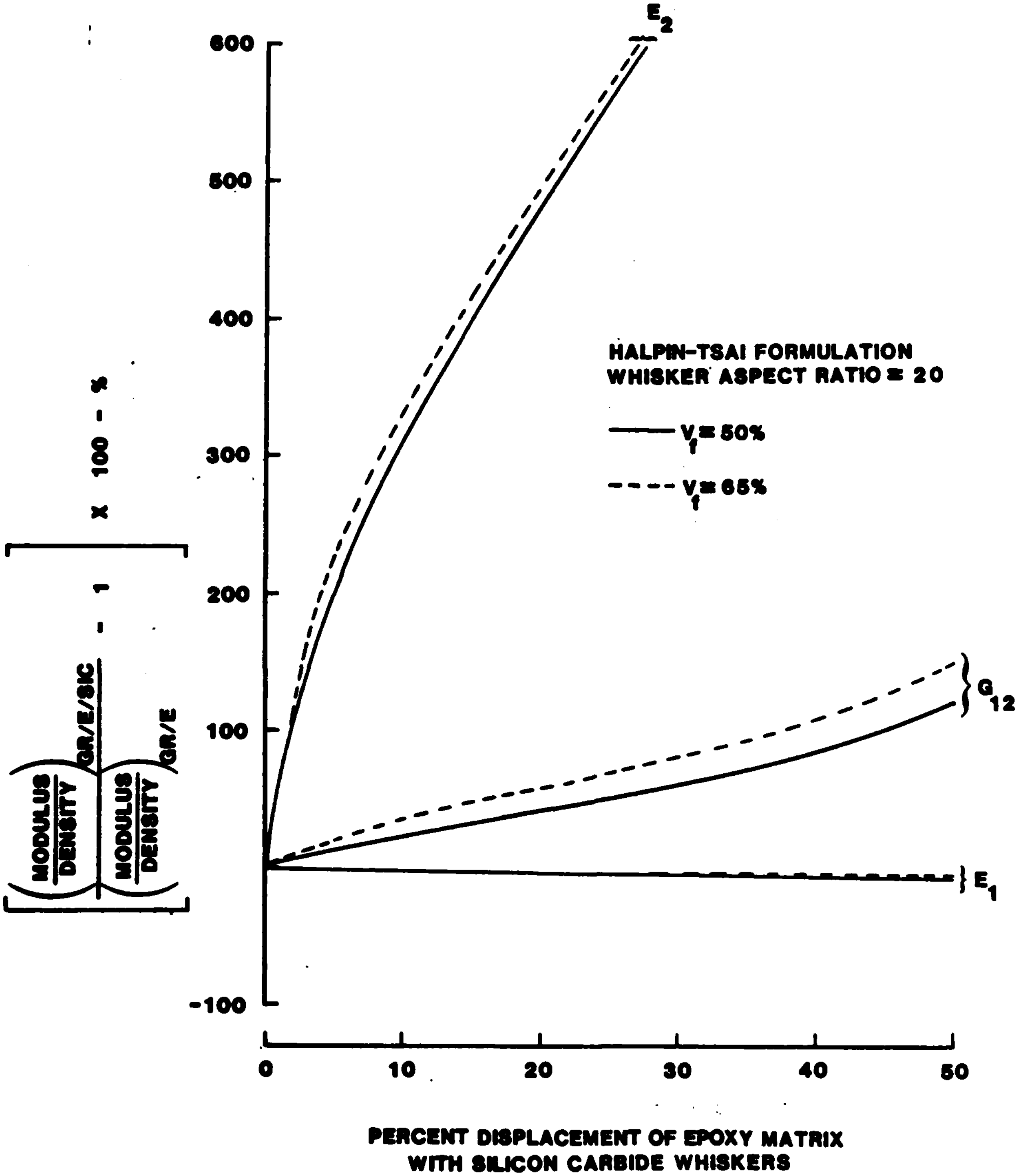


FIGURE 10. PROJECTED MODULUS IMPROVEMENTS DUE TO THE ADDITION OF SIC WHISKERS: HALPIN-TSAI FORMULATION WITH WHISKER ASPECT RATIO EQUAL TO 20.

TABLE 4. CALCULATED MODULI USING HALPIN-TSAI/PAUL FORMULATION.

$V_f/V_E/V_{SiC}$	ρ (LBS/IN^3)	E_1 (10^6PSI)	E_2 (10^6PSI)	G_{12} (10^6PSI)
50/50/0	.0545	17.32	1.27	.68
50/47.5/2.5	.0563	17.50	3.53	1.06
50/45/5	.0580	17.59	4.08	1.23
50/42.5/7.5	.0598	17.67	4.59	1.43
50/37.5/12.5	.0633	17.85	5.57	1.80
50/32.5/17.5	.0669	18.06	6.68	2.23
50/25/25	.0722	18.51	8.75	3.21
65/35/0	.0576	22.32	1.79	1.04
65/33.25/1.75	.0588	22.45	5.41	1.61
65/31.5/3.5	.0601	22.51	6.19	1.88
65/29.75/5.25	.0614	22.57	6.89	2.18
65/26.25/8.75	.0639	22.69	8.21	2.75
65/22.75/12.25	.0663	22.84	9.65	3.41
65/17.5/17/5	.0701	23.16	12.20	4.89

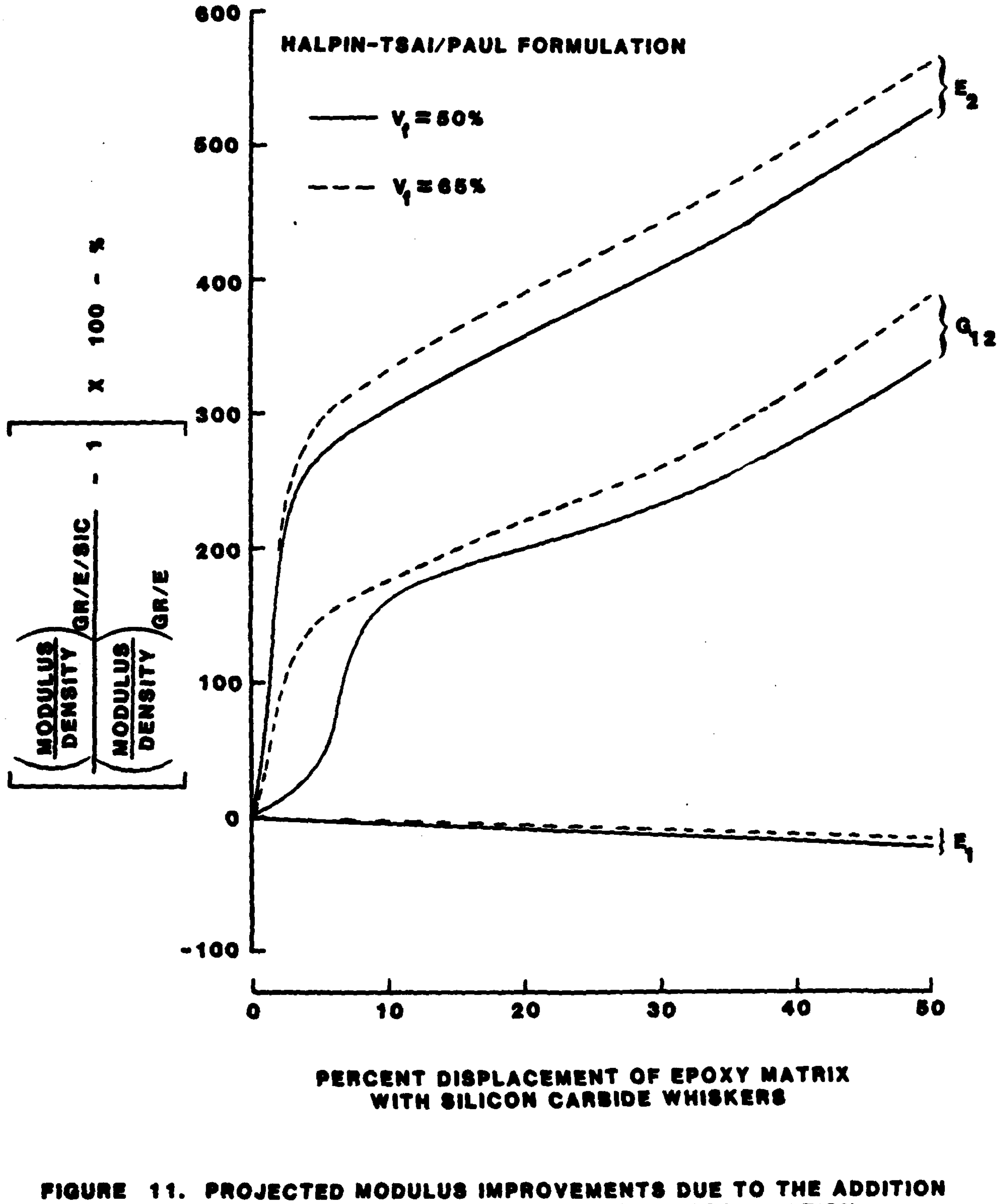


FIGURE 11. PROJECTED MODULUS IMPROVEMENTS DUE TO THE ADDITION OF SIC WHISKERS: HALPIN-TSAI/PAUL FORMULATION.

interleaf between the borsic filaments.

2) An increase in transverse tensile modulus from 15 to 20 $X10^6$ PSI has been achieved in the above ranges.

3) Incorporation of 15% V_{SiC} β-SiC into the interleaf between the Borsic filaments have resulted in the improvement of the elevated temperature tensile strength. No loss in strength occurs up to 400°F, (i.e., the increase in tensile strength due to whisker addition was completely retained at temperatures up to 400°F).

4) At 600°F, a 30% V_f Borsic composite, reinforced with a β-SiC reinforced interleaf, has a transverse tensile strength of 27.5 KPSI as compared to 2.48 KPSI for unreinforced 6061 aluminum.

The material(s) selected for third-phase reinforcement must meet the criteria listed in Section 2.2. Candidate materials must show specific stiffness improvements using the procedures given in Section 2.5 and illustrated above. In addition to the specific stiffness calculations, projections can be made of other properties, such as strength and moisture absorption (where possible). Since the major emphasis of this report is to present a technical review, projected changes in other material properties are not presented.

4.0 CONCLUDING REMARKS

Two methods of improving the matrix dominated properties of composite materials have been reviewed. In the fiber carrier method, silicon carbide whiskers are grown on the surface of the fibers. Any fiber which can withstand the furnace whiskerizing temperature of 1480°C can be used. These whiskered fibers can then be used to fabricate prepreg material. In the matrix carrier method, particulates or whiskers are mixed into the resin prior to prepreg fabrication. Any type of fiber may be used in this method. However, the size of the particulate or whisker is dependent on the continuous fiber diameter and fiber volume fraction desired.

Both methods have the potential to increase the matrix dominated properties of the composite. Since neither method has been explored in recent times, it is recommended that:

1) A feasibility study should be initiated to examine the property improvement potential of each method.

2) The feasibility studies should compare the properties measured from a Gr/E/SiC composite, for example, to those properties measured from a control Gr/E composite. (See Appendix for recommended mechanical property tests).

3) Theoretical predictions of property improvements should be verified where possible.

4) A scanning electron microscopic study should be conducted to examine microstructure and failure modes.

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APPENDIX A

RECOMMENDED FEASIBILITY STUDY TEST METHODS

Graphite/epoxy/whisker and/or graphite/epoxy/particulate composite materials can be manufactured to examine various properties. The data generated for these three-phase composite materials can be compared to analytical predictions, where possible, and to control specimens. Control specimens should consist of graphite/epoxy having the same graphite fiber volume fractions as the three-phase, composite material specimens.

The limited test data to be generated using the specimens described here are to demonstrate proof-of-concept in the recommended feasibility study. If the results of the recommended feasibility study are favorable, then subsequent studies can be used to examine additional properties, environmental conditions, materials, etc.

A. TENSION

The specimen configuration illustrated in Figure A.1 can be used to determine E_1, ν_{12}, and ultimate tensile strength in the fiber direction. The specimen configuration illustrated in Figure A.2 can be used to determine E_2, ν_{21}, and ultimate tensile strength transverse to the fibers.

B. COMPRESSION

Compression modulus and strength can be measured in the longitudinal and transverse directions using the IITRI Specimen (Reference A1)* illustrated in Figure A.3. The restrained and unrestrained block compression test specimen (Reference A2) illustrated in Figure A.4 can be used to examine changes in compressive failure modes.

* Reference at end of Appendix

C. SHEAR

The Rail Shear Specimen (Reference A3), illustrated in Figure A.5, can be used to measure G_{12} and ultimate shear strength.

D. INTERLAMINAR

Interlaminar shear stress can be examined using the Short Beam Shear (Reference A4) and Multi-span Beam Shear (Reference A5) Specimens illustrated in Figures A.6 and A.7, respectively. Interlaminar normal stress can be examined using the specimen illustrated in Figure A.2, with a $[\pm 15, \pm 45]_s$ layup (References A6, A7).

E. ENVIRONMENTAL

Moisture absorption can be examined using the specimen illustrated in Figure A.8. Additional specimens of this type can be used to measure the coefficients of thermal expansion in the longitudinal and transverse directions.

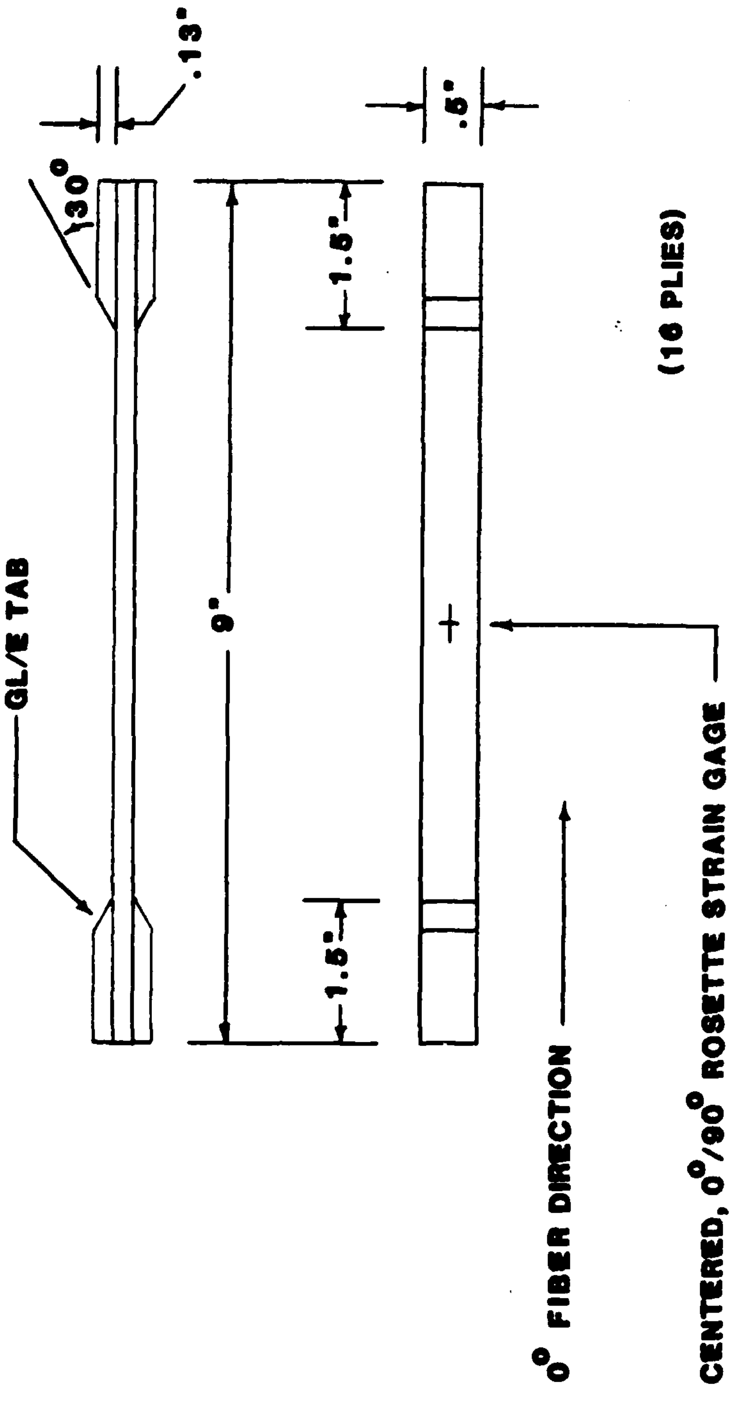

FIGURE A1. LONGITUDINAL TENSION SPECIMEN.

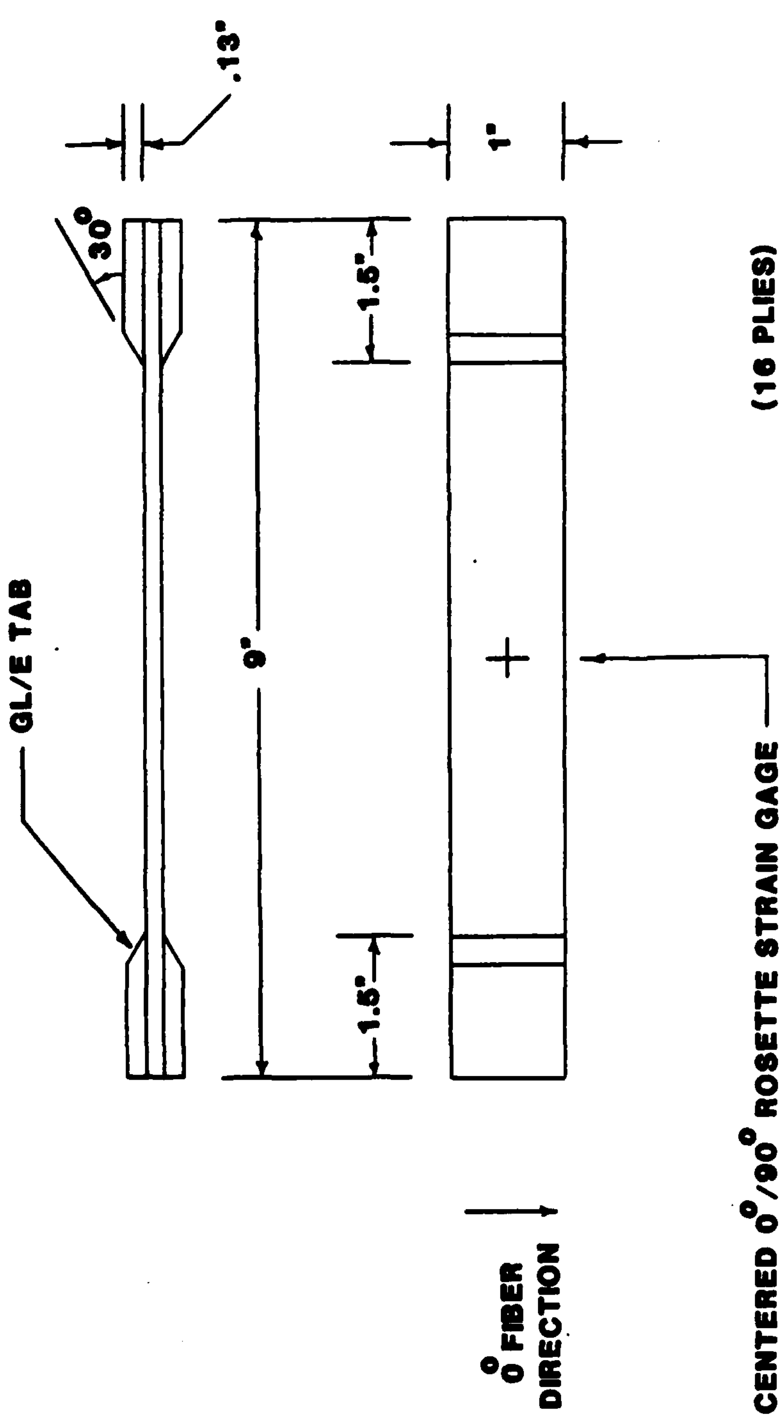

FIGURE A2. TRANSVERSE TENSION SPECIMEN.

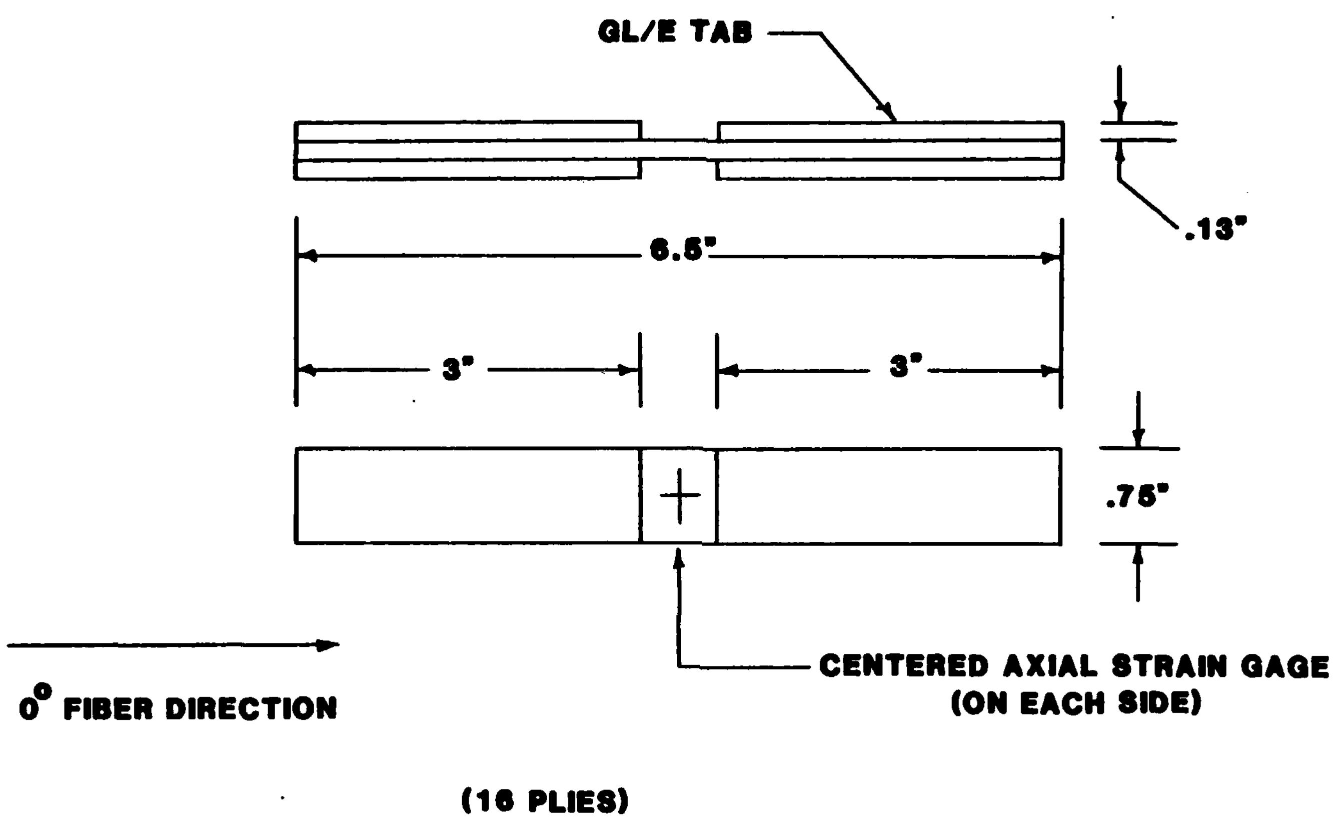

FIGURE A3. LONGITUDINAL AND TRANSVERSE IITRI COMPRESSION SPECIMEN.

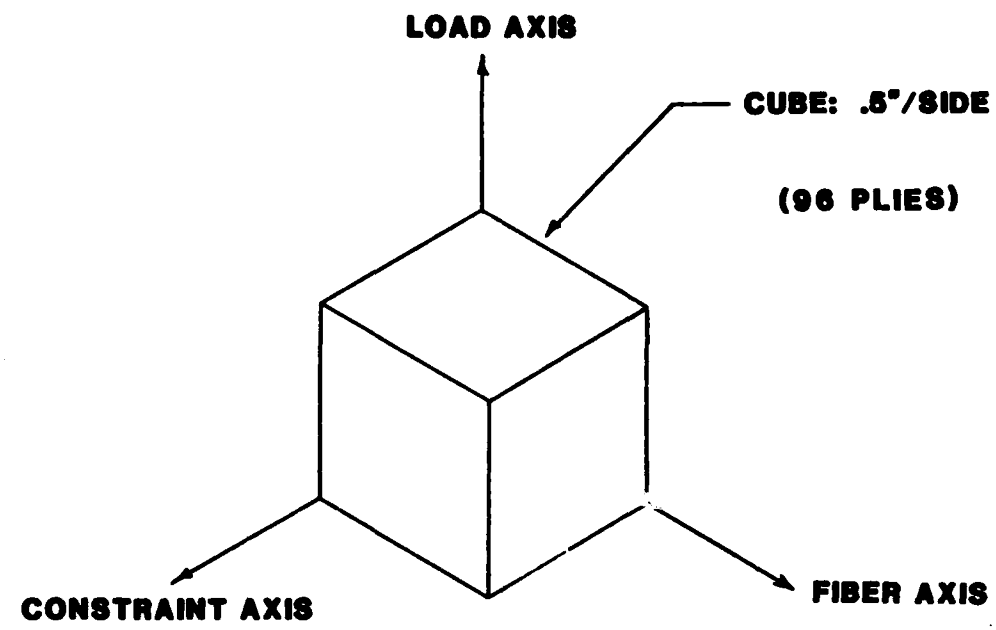

FIGURE A4. BLOCK COMPRESSION TEST SPECIMEN.

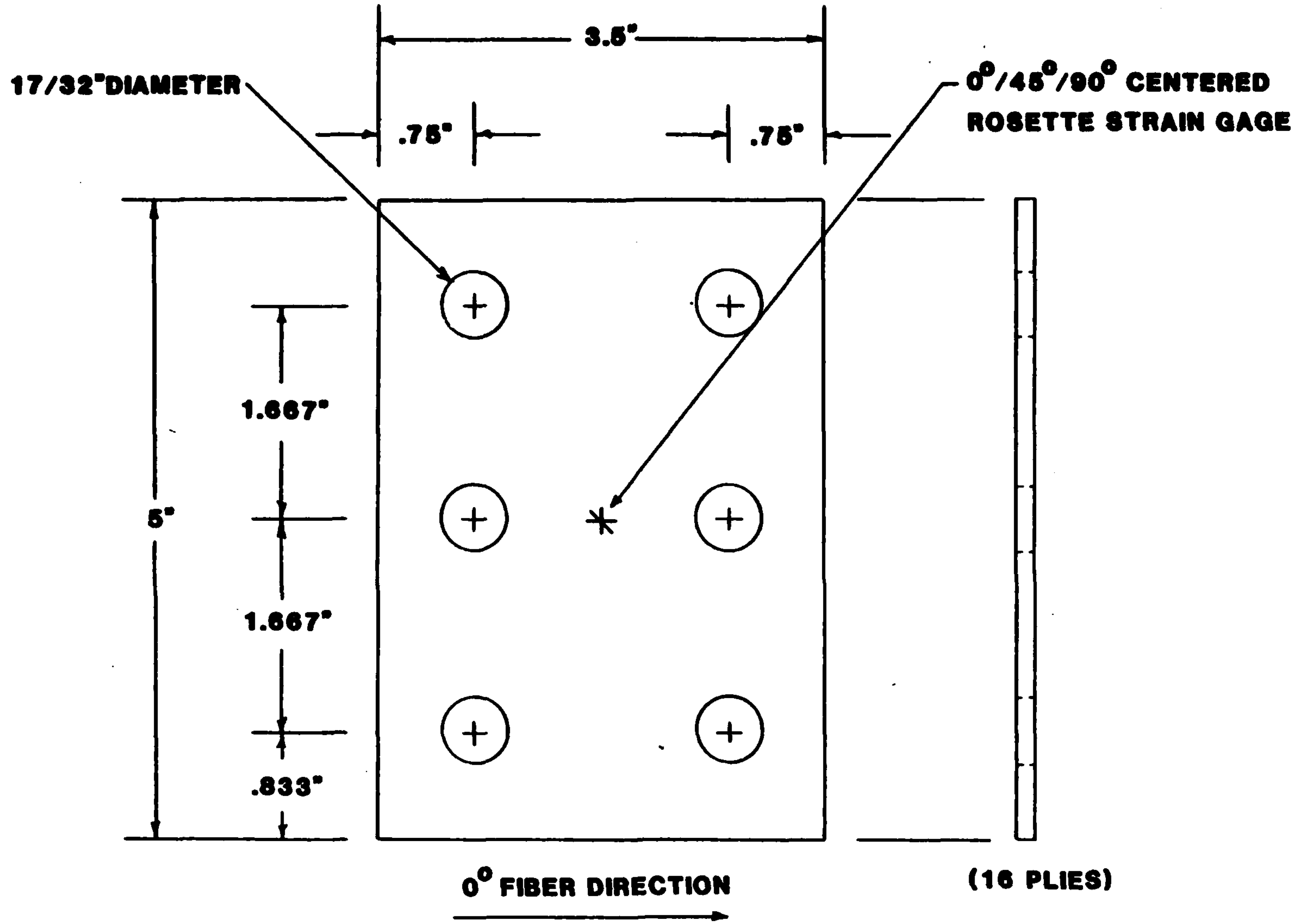

FIGURE A5. RAIL SHEAR TEST SPECIMEN (LOAD INTRODUCTION RAILS NOT SHOWN).

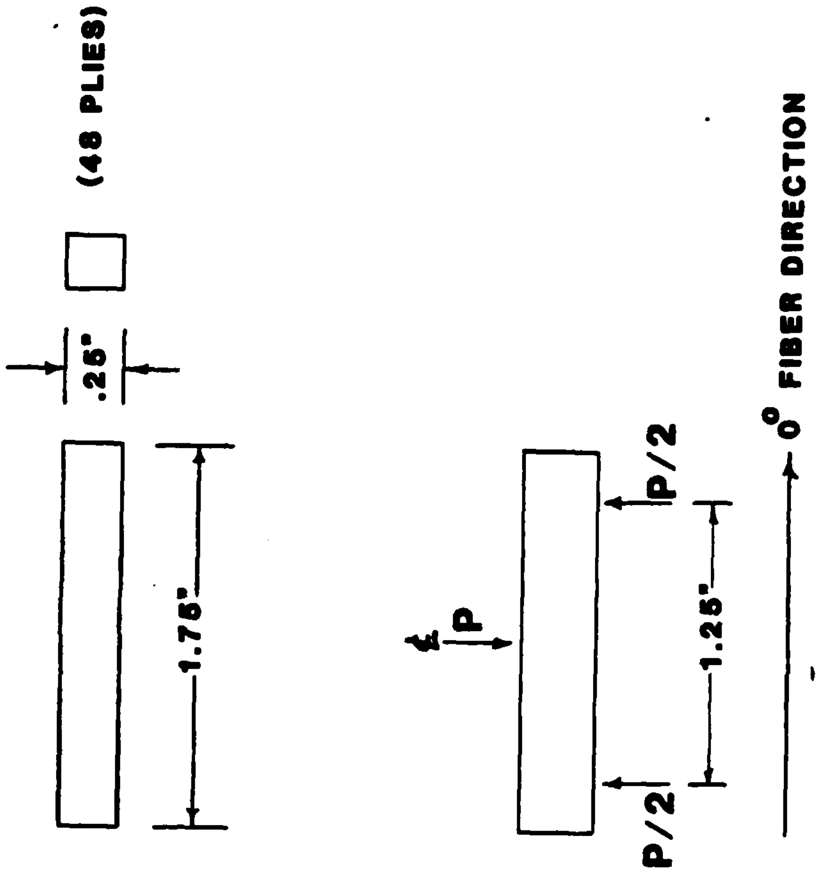

FIGURE A6. SHORT BEAM SHEAR TEST SPECIMEN.

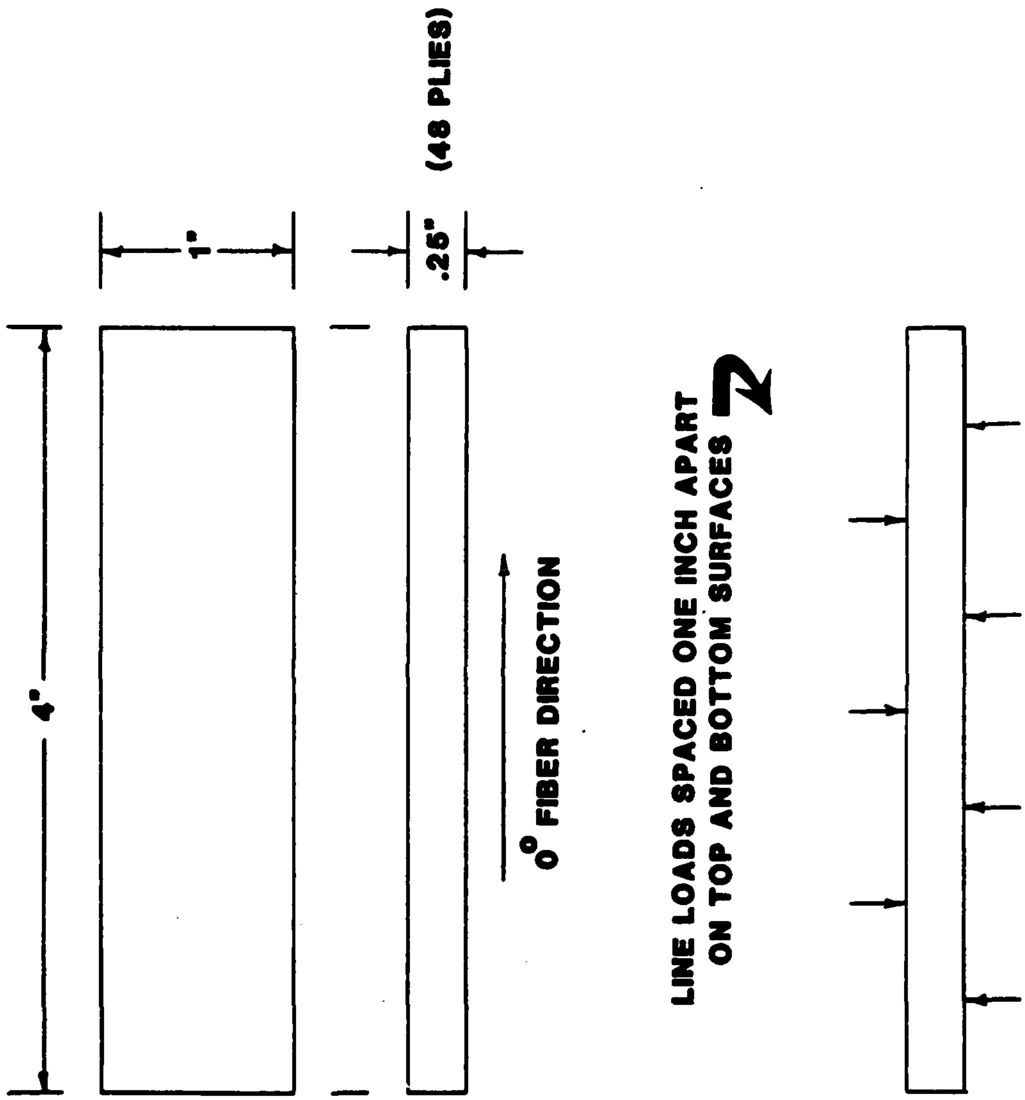

FIGURE A7. MULTI-SPAN BEAM SHEAR TEST SPECIMEN.

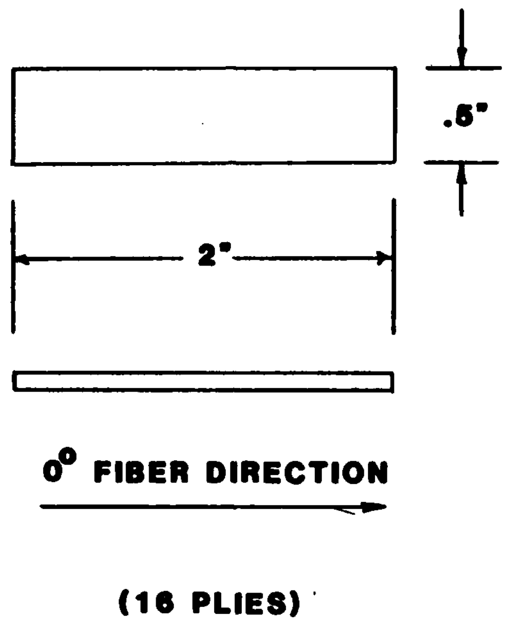

FIGURE A8. SPECIMEN FOR DETERMINING COEFFICIENTS OF THERMAL EXPANSION AND MOISTURE ABSORPTION RATES.

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